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THE PRODUCTION OF ATOMIC OXYGEN BY THE THERMAL DECOMPOSITION OF OZONE

by

John R. Kelso

June 1966

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THE PRODUCTION OF ATOMIC OXYGEN BY THE
THERMAL DECOMPOSITION OF OZONE

John R. Kelso

Interior Ballistics Laboratory

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DEFENSE ATOMIC SUPPORT AGENCY

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June 1966

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THERMAL DECOMPOSITION OF OZONE

ABSTRACT

To study the reactions of atomic oxygen it is desirable to have a supply that is free of metastable energetic species of molecular and atomic oxygen which are formed in discharged oxygen and because of their side reactions can lead to serious errors in calculations. Therefore, the homogeneous, thermal, gas phase decomposition of dry ozone was tried and found to be a satisfactory source if the residence time of ozone in the furnace is carefully regulated by furnace length and carrier gas flow, followed by rapid cooling of the products of decomposition.

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TABLE OF SYMBOLS

\AA	angstrom
ac	alternating current
cm	centimeter
cm^3/mole	cubic centimeter per mole
$^{\circ}\text{C}$	degree centigrade
diam	diameter
kcal	kilocalorie
kV	kilovolt
$^{\circ}\text{K}$	degree Kelvin
ml	milliliter
mm	millimeter
ml/min	milliliter per minute
msec	millisecond
mtorr	micron
[M]	concentration of M, moles per liter
N.T.P.	normal temperature and pressure
sec^{-1}	per second
torr	millimeter

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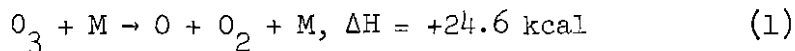
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INTRODUCTION

During the past several years many studies of chemical reactions^{1,2,3*} and recombination rates^{4,5,6} of atomic oxygen have been made in this laboratory for a better understanding and prediction of the reactions occurring in the upper atmosphere with regard to 1) re-entry problems, 2) blackout, and 3) other military applications. Throughout most of this work atomic oxygen was produced in an electrodeless discharge excited by microwave radiation at 2450 Mc/sec, generated by a Magnetron (Raytheon type QK-390) operated at input powers up to 800 watts. Although this has been the universally accepted manner of producing atomic species of gases by many workers the O-atom production in extremely pure O₂ is quite low (.1 to .2% of total O₂ flow through the discharge) and recent work^{4,7,8} has shown that metastable energetic species of O₂ are also generated in the discharge such as O₂[¹Σ_g, ¹Δ_g, ³Σ_g]. Under these conditions, then, erroneous results are obtained if the presence of these species is not taken into account.

One source should clearly be the homogeneous, thermal gas phase decomposition of ozone⁹,



whereby a mole of atomic oxygen could be obtained for each mole of ozone decomposed. Ozone is easily produced, easily stored, and decomposes at a comparatively low temperature.

This report is a continuation and improvement on a method first used in work⁴ published earlier.

* *Superscript numbers denote references which may be found on page 25.*

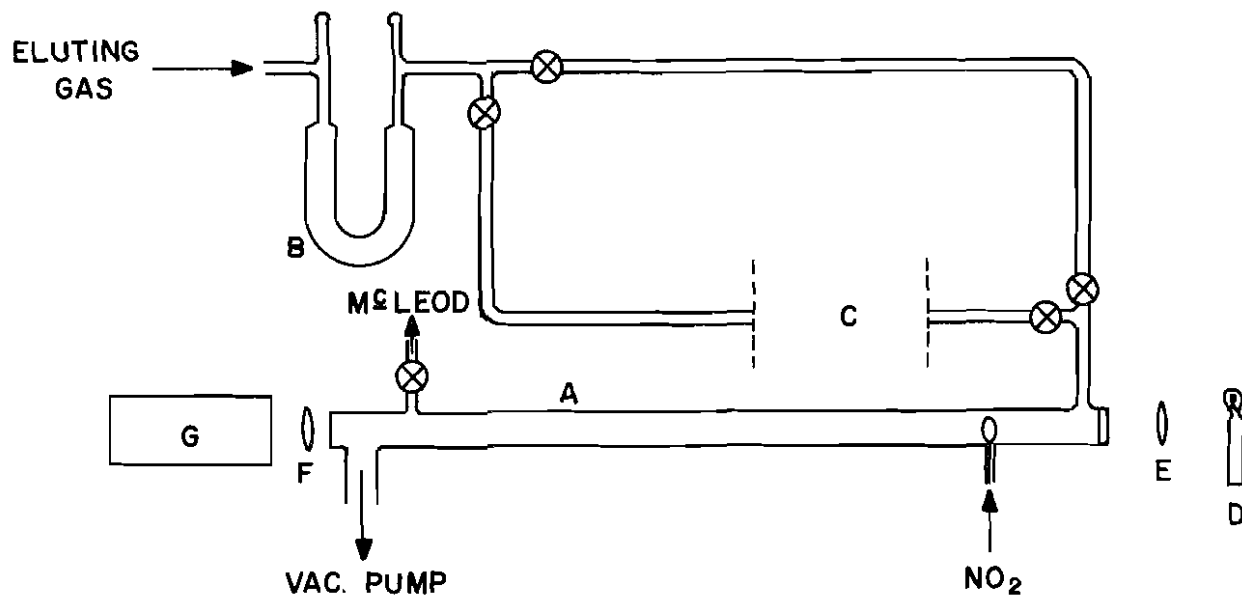
THE EXPERIMENT

The ozone was prepared from thoroughly dried cylinder O_2 (Southern) by first passing the O_2 at one atmosphere through a quartz tube, 1 cm in diameter, 30 cm long, packed with quartz chips, and heated to $1100^{\circ}C$, then through a column 90 cm long packed with zeolite molecular sieve, type 5A, to assure the removal of hydrogenous impurities.

The purified O_2 , still at atmospheric pressure, then flowed through the annular space of a Siemens-type ozonizer across which an ac voltage of 10-12 kv produced 0.4 - 3% ozone. The resulting mixture of ozone and oxygen then flowed into a trap containing 600 grams of silica gel (Davison, 6-12 mesh) which was cooled to $-78^{\circ}C$, and upon which the O_3 was adsorbed to a density of loading of 5-8%, which represents 30 to 48 grams of ozone. At this temperature the O_3 partial pressure over silica gel is 3 torr¹⁰. Higher O_3 pressures are obtained by raising the silica gel temperature above $-78^{\circ}C$. Passing a carrier gas over the gel for O_3 elution one obtains a constant flow of O_3 plus carrier gas until the density of loading drops off.

A major requirement was a means of measuring the concentration of the O_3 before and after its decomposition by thermal means. This analysis system (Fig. I) consists of a Pyrex glass tube 115 cm long, equipped with a quartz window at each end, and connected to a vacuum pump. Light emitted from a mercury lamp (Spectroline quartz Pencil Lamp) placed at one end of the tube was collimated by a suitable quartz lens, passed through light filters to isolate the 2537 Å line as described by Kasha¹¹ and hence through the 115 cm path length to a quartz condensing lens and a 1P28 photomultiplier tube. The output of the photomultiplier was amplified and displayed on a Leeds and Northrup recorder. In a later system, the filters were replaced by a small grating monochromator (Farrand, No. 103420). The minimum analyzable O_3 pressure was about $0.5 - 1 \times 10^{-4}$ torr.

FIGURE I. DIAGRAM OF APPARATUS



- A. PYREX TUBE, 115 CM LONG, 2.54 CM ID, WITH QUARTZ WINDOWS
- B. O₃ TRAP
- C. O₃ DECOMPOSITION FURNACE
- D. MERCURY LIGHT SOURCE
- E. QUARTZ COLLIMATING LENS
- F. QUARTZ CONDENSING LENS
- G. MONOCHROMATOR

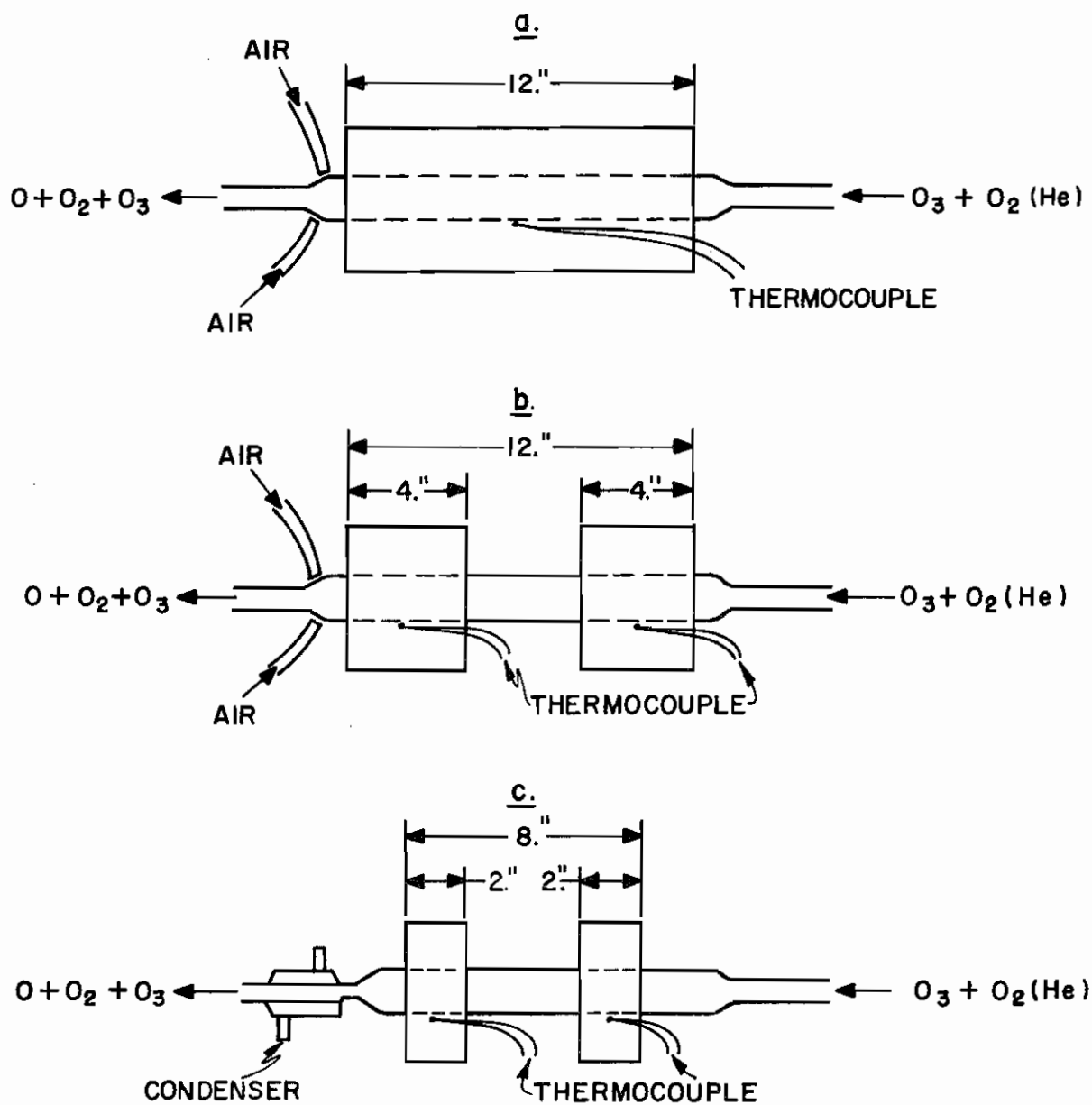
The $[O]$ was determined at the downstream end of the decomposition furnace by the "gas titration" method^{12,13} using nitrogen dioxide.

Not knowing what residence times in the decomposition furnace would be most advantageous to produce a maximum concentration of atomic oxygen by the complete decomposition of O_3 , the decision to give the O_3 ample time for decomposition was adhered to. For the first attempt a muffle furnace (Figure IIa) with a heating section large enough to accommodate a quartz reaction tube 30 cm long, 2.5 cm internal diameter was used. Two jets of compressed air were placed at the downstream end of the reaction tube to cool the resultant gas mixture. The furnace was fitted with a platinum, platinum-rhodium thermocouple for temperature measurement. The furnace and its accompanying quartz tube were connected to the O_3 analysis system and a by-pass was installed around the furnace to allow a measurement of $[O_3]$ before and after decomposition.

To obtain shorter residence times in the heated section for O_3 decomposition, two smaller furnaces, each of 4 inch length were constructed. Alundum tubing, $1\frac{1}{4}$ inch ID, was first wound with a layer of asbestos, then with coiled "Nichrome" heating wire with asbestos cord spacers to prevent the individual coils from shorting out, and finally coated with cement (Johns-Manville Refractory Cement, No. 20). Each furnace had a resistance of 14 ohms. The two sections were then mounted in series on a piece of quartz tubing 25 cm long and 2.5 cm OD with approximately 1 cm spacing between them. Thermocouples were placed between the inner wall of the furnace and the outer wall of the quartz tube. The downstream end of the tubing was again cooled by air jets, and the whole assembly (Figure IIb) fitted into the space previously filled by the original furnace.

As will be noted in the discussion, still shorter residence times were advisable; therefore, two more furnaces each of 2 inch length were constructed in the same manner as the 4 inch furnaces and mounted in series on a piece of 1 inch OD quartz tubing (Fig. IIc) for emplacement in the same position within the flow system. For more efficient

FIGURE II
DIAGRAM OF THREE TYPES OF FURNACES



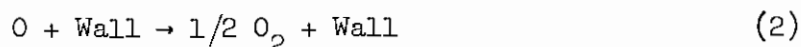
cooling of the decomposed gas mixture a quartz water cooled condenser was placed downstream of the heated portion as close to the final heater as was dared, considering the extreme thermal shock to the tubing induced by heating to 1000°C followed by immediate cooling. It was found that a short space of 1.25 cm was tolerable and desirable.

The procedure used during the heating period followed the same pattern with all furnaces tried. With the furnace at room temperature total flows of O_3 with O_2 as carrier gas (He in some cases for comparison only) of 30 to 600 $\text{Ml (N.T.P.)}/\text{min}$ were allowed to flow first through the by-pass into the evacuated O_3 analysis system and then through the furnace where the decomposition occurred while the $[\text{O}_3]$ was being monitored. Several experiments showed that, as the temperature of the O_3 decomposition furnace was raised, the $[\text{O}_3]$ remained unchanged until a temperature of 500°C was reached, then decreased with further temperature increase so that at 750°C and above, the O_3 was completely decomposed. At most flow rates and at all pressures a small amount of O_3 (henceforth referred to as residual O_3) was found to be present even at the highest furnace temperature. This was undoubtedly due to the recombination of O with O_2 in the cool portion of the system.

RESULTS AND DISCUSSION

Following the experimental procedure described earlier using the 12 inch long furnace, results were obtained which were quite disappointing. The yield of atomic oxygen was extremely low ($\sim 2\%$ of the ozone input) and a very small quantity of residual ozone was noted. This was puzzling when one considers the half-life of O_3 is about 1 msec at 1000°C at a pressure of 3 torr (calculated assuming k for the reaction to be $1.94 \times 10^{10} \text{ cm}^3/\text{mole sec}$).

Previous studies in this laboratory¹⁴ have shown that the rate of the heterogeneous O-atom recombination -



on hot vycor walls is fast, and increases with increasing temperature. At 1090°K , for example, the rate constant is 21.3 sec^{-1} . This can explain, in part, the low $[O]$. Considering the length of this furnace, which allowed residence times of 10-80 msec depending on the flow rate of carrier gas, there was ample time for this undesirable reaction to occur.

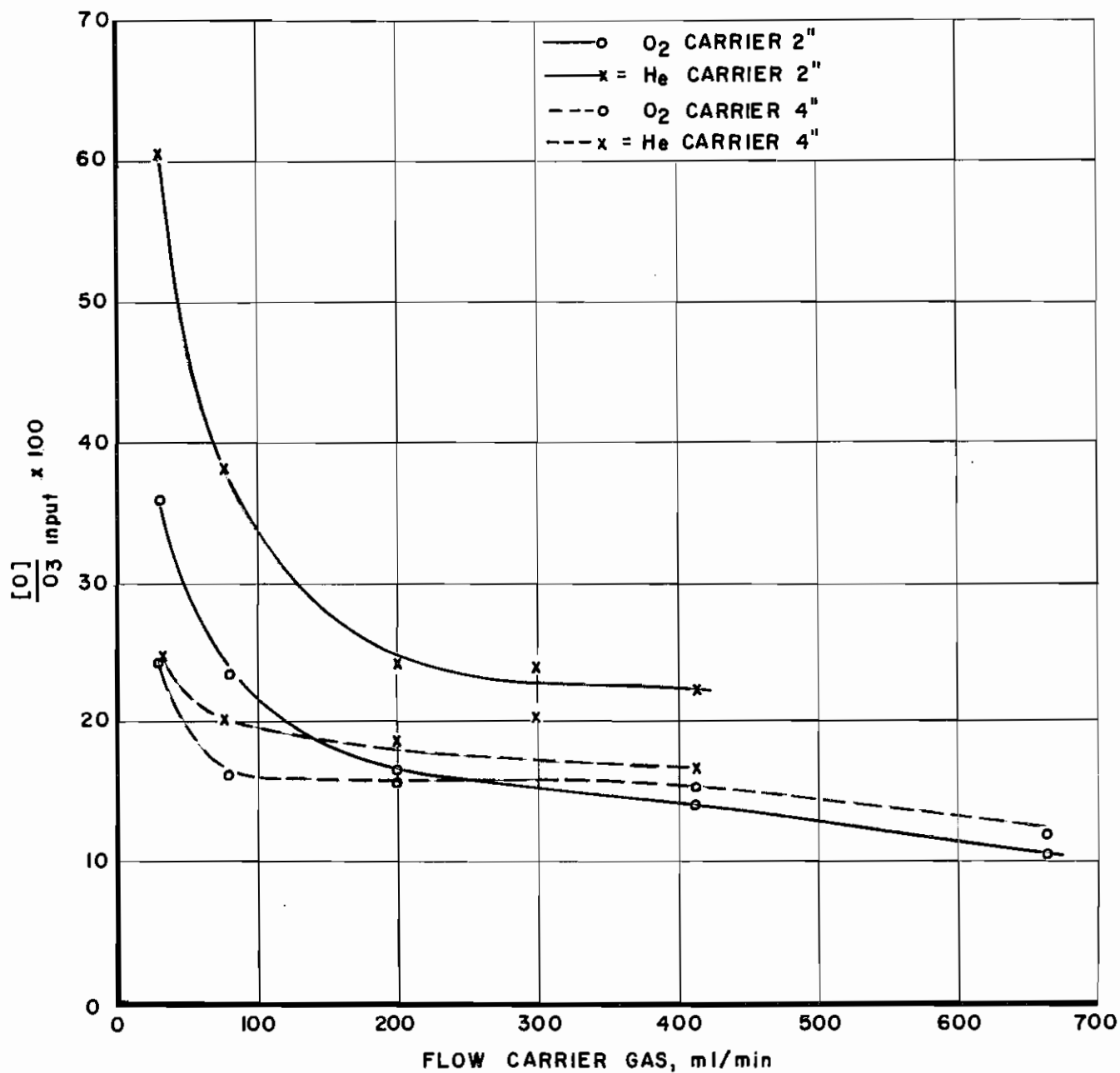
Another possible undesirable reaction that should be considered is the homogeneous gas phase reaction -



which would not only lessen the anticipated $[O]$, but would give the residual $[O_3]$ encountered. However, this reaction could only be taking place in the cool portion of the system downstream from the furnace because in the heated section its equilibrium is strongly in favor of the reverse. If the carrier gas is O_2 (as was used here) then the reaction would have a good chance of occurring.

The next phase of the experimentation was dictated by the urgent need to reduce the residence times in the heated zone to curtail reaction (2). Two 4-inch furnace sections were used initially; however, it was soon found that the use of only one 4-inch section was

FIGURE III - PLOT OF % O OF O_3 INPUT FOR COMPARISON
OF EFFICIENCY OF 1-2" FURNACE WITH 1-4"
FURNACE USING O_2 OR He AS CARRIER GAS



most advantageous, because a higher $[O]$ was obtained than with the operation of the two in series. As shown in Table II, with operation of one furnace section at slightly over 1000°C , and at total flow rates of 30 ml(N.T.P)/min the percentage of atomic oxygen obtained is equal to 24.4% of the total ozone input, in comparison with 15.4% obtained with the operation of both furnaces at similar flow conditions (Table I). It can also be noted that increasing the flow rates of oxygen as the carrier 1) increased the atomic oxygen concentration, though the apparent percentage of ozone decomposed fell off; and 2) increased the residual O_3 [due to reaction (3) in the cool section].

To prove that reaction (3) was occurring, helium was substituted for O_2 as carrier gas. The rates of chemical reactions are strongly dependent upon the concentrations of the reactants, and in this case, complete removal of one of the reactants should prevent the reaction. However, reaction (2) is constantly supplying O_2 , although the $[O_2]$ derived from this reaction is very low in comparison to the $[O_2]$ when using O_2 as carrier. As had been expected, the $[O]$ under similar conditions of total flow rate did increase (Table II) and resulted in a higher percentage of atomic oxygen. Likewise, the residual ozone concentration remained at a much lower level. One would not expect reaction (2) to be changed, as at the temperatures of the furnace no reaction occurs between O_2 and O .

It should be mentioned here that the apparent lack of conformity of the residual $[O_3]$ noted in Table II can only be ascribed to experimental technique, and is not alarming when one considers the extremely small quantities of ozone in question.

The pair of 2-inch long furnaces was next installed and an increase of percentage of O was immediately noted over the former experimental set-ups and again the greatest increase was found while operating a single furnace section (Tables III and IV). An assumption had been made here that perhaps at the highest flow rates of carrier (shortest residence times) the O_3 decomposition would not have had

TABLE I

Two 4-Inch Furnace Sections Operating. $T = 1015^{\circ}\text{C}$ O_2 elution of O_3

P	P_{total} , torr	$F_{\text{carrier, O}_2}$ ml/min	F_{O} ml/min	O_3 input p, mtorr	residual O_3 p, mtorr	$\frac{F_{\text{O}}}{\text{O}_3 \text{ input}} \times 100$	$\frac{F_{\text{O}}}{F_{\text{O}_2}} \times 100$
	0.49	30	0.188	19.8	0.0	15.4	.6
	0.91	80	0.310	30.3	0.0	11.6	.3
18	1.41	200	0.492	31.7	0.0	7.7	.2
	2.52	535	1.17	78.7	0.0	7.0	.2

TABLE II

One 4-Inch Furnace Section Operating, $T = 1015^{\circ}\text{C}$

	P_{total} , torr	$F_{\text{carrier, O}_2}$ ml/min	O_2 elution of O_3		$\frac{F_{\text{O}}}{F_{\text{O}_3 \text{ input}}} \times 100$	$\frac{F_{\text{O}}}{F_{\text{O}_2}} \times 100$
			F_{O} ml/min	O_3 input p, mtorr	residual O_3 p, mtorr	
61	0.555	30	0.164	12.4	0	24.3
	0.80	80	0.286	17.7	0.2	16.1
	1.35	200	0.638	27.4	0.0	15.7
	2.15	415	1.474	49.6	0.3	15.4
	2.88	65	2.041	73.9	1.0	11.9
<u>He elution of O_3</u>						
	0.610	31	0.225	15.4	0.1	28.6
	0.93	80	0.421	24.1	0.0	20.2
	1.63	200	1.034	45.1	0.0	18.6
	1.86	300	1.490	52.1	0.0	17.7
	2.19	415	2.216	69.1	0.0	16.1

TABLE III

Two 2-Inch Furnace Sections Operating, $T = 1015^{\circ}\text{C}$

	P_{total} , torr	$F_{\text{carrier, O}_2}$ ml/min	F_{O} ml/min	O_2 elution of O_3		$\frac{F_{\text{O}}}{\text{O}_3 \text{ input}} \times 100$	$\frac{F_{\text{O}}}{F_{\text{O}_2}} \times 100$
				O_3 input p, mtorr	residual O_3 p, mtorr		
20	.555	30	.164	12.4	0.0	24.4	0.5
	.80	80	.286	17.7	.2	16.6	0.3
	1.35	200	.638	27.4	.0	15.7	0.3
	2.15	415	1.474	49.6	.3	15.4	0.3
	2.88	665	2.041	73.9	1.0	11.9	0.3
He elution of O_3							
		$F_{\text{carrier, He}}$ ml/min					
	.605	32	.220	15.4	.1	27.0	
	.90	78	.415	23.9	0.0	20.0	
	1.59	200	1.068	44.8	.1	18.9	
	1.82	300	1.676	50.2	0.0	20.3	
	2.18	415	2.116	67.1	.1	16.5	

TABLE IV

One 2-Inch Furnace Section Operating, $T = 1015^{\circ}\text{C}$

P_{total} torr	$F_{\text{carrier, O}_2}$ ml/min	<u>O₂ elution of O₃</u>				
		F_{O} ml/min	O ₃ input p, mtorr	residual O ₃ p, mtorr	$\frac{F_{\text{O}}}{\text{O}_3 \text{ input}} \times 100$	$\frac{F_{\text{O}}}{F_{\text{O}_2}} \times 100$
0.70	30	0.267	17.3	0.3	36.0	.8
0.93	80	0.424	20.9	0.7	23.5	.5
1.355	200	0.905	37.5	0.6	16.4	.4
2.18	415	1.899	68.6	0.8	14.5	.4
2.92	665	2.320	96.4	1.7	10.6	.3
<u>He elution of O₃</u>						
	$F_{\text{carrier, He}}$ ml/min					
0.87	32	0.415	18.6	0.8	60.6	
1.18	78	0.672	26.8	0.4	38.0	
1.625	200	1.575	52.6	0.4	24.3	
1.820	300	2.258	57.1	0.1	24.0	
2.200	415	3.150	75.0	0.1	22.3	

time to go to completion. This was not the case and indicates that still shorter residence times would increase the O-atom yield. Upon substitution of He for O_2 as carrier, the expected higher percentage of O based on the O_3 input was obtained (hence added proof of the occurrence of reaction (3)). However, note that residual O_3 in Table IV with He elution is highest at the lowest pressure and decreases with increasing pressure indicating that O_3 decomposition may not have been complete at lower pressures.

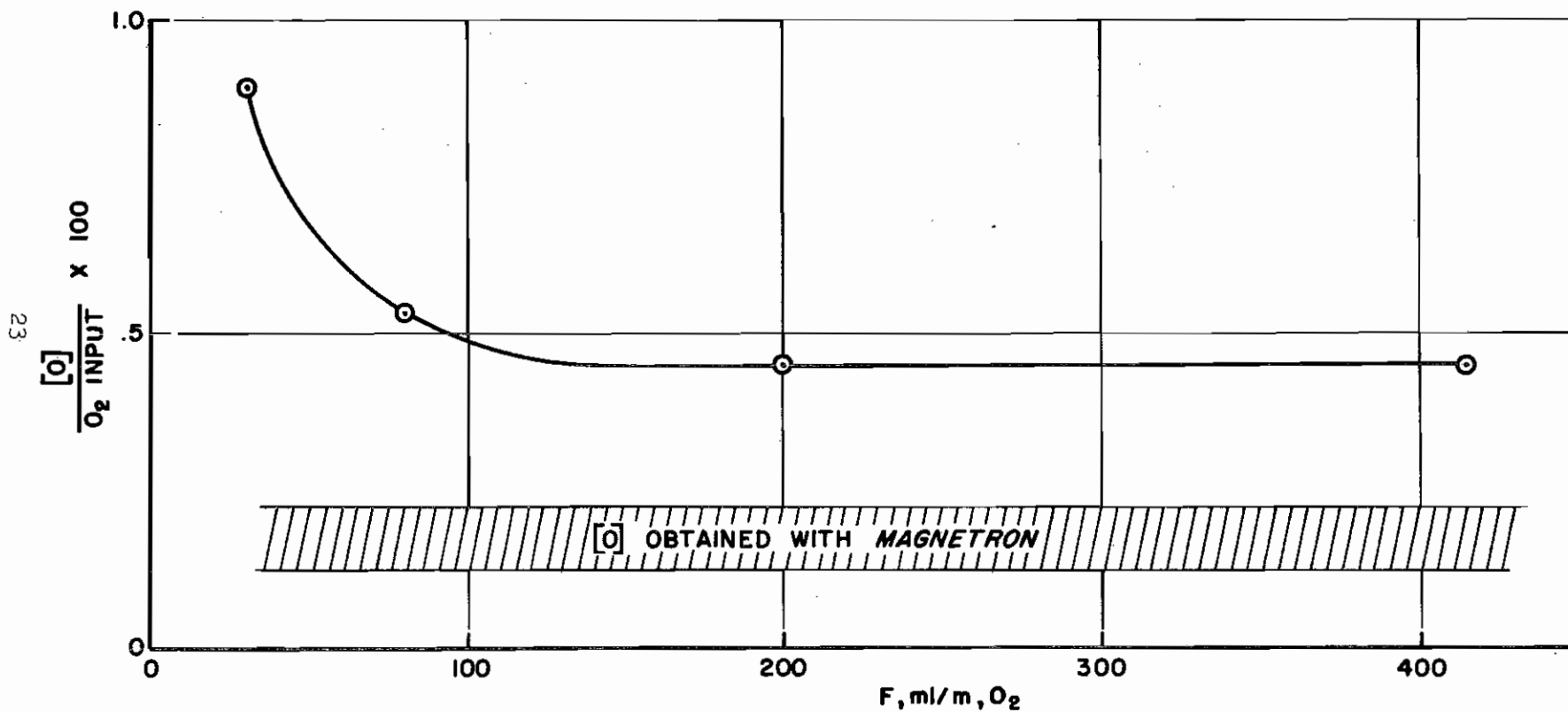
SUMMARY

These findings show that the thermal, homogeneous, gas phase decomposition of ozone is a suitable source of atomic oxygen. Figure IV presents a plotted comparison between the oxygen atom concentration obtained by the carefully controlled decomposition of ozone and by a microwave discharge in extremely pure, dry oxygen. During the heating or decomposition period, the choice of either O_2 or He as the carrier is not important, and the major problem to be dealt with is the fast recombination of O on the hot wall. In the cool portion of the system, the major losses of O occur by the slow reaction of O with O_2 . Therefore, the hot wall recombination of O becomes the limiting factor.

The method does carry certain limitations. 1) a high concentration of O_3 can be obtained from the trap only while the density of loading stays constant (throughout the first half of the total elution). During a long run (a few hours) one would begin to notice a gradual decrease in [O]. However, this can be controlled by anticipating the quantity required beforehand and constructing a suitably large trap. 2) The residual $[O_3]$ might be undesirable in some applications, as well as the presence of O_2 or N_2 as used in this work for carrier gas.

A further study is being considered, that is, the thermal decomposition of O_3 by means of a heated filament placed as near as possible to the location within the system where the atomic oxygen is to be used to prevent the occurrence of reaction (4), and to keep the

FIGURE IV - COMPARISON OF $[O]$ OBTAINED BY THERMAL DECOMPOSITION
OF O_3 IN A 2 INCH FURNACE AND BY MICROWAVE
RADIATION OF DRY, PURE O_2 .



vydor walls in the vicinity of the filament suitably cooled to prevent the occurrence of reaction (3). One such attempt was actually made using a platinum wire (.02 inch diam.) filament. Upon being heated to a bright red heat the filament collapsed and was immediately unfit for further use. Perhaps the use of a rhodium filament would prevent this difficulty. However, great care should be taken to promote an extremely short contact time with the filament (for decomposition of O_3) as the hot metal surfaces are excellent for the recombination of O-atoms.

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JOHN R. KELSO

* Formerly Chief, Chemical Physics Branch, IBL: now at the University of Pittsburgh

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13. ABSTRACT To study the reactions of atomic oxygen it is desirable to have a supply that is free of metastable energetic species of molecular and atomic oxygen which are formed in discharged oxygen and because of their side reactions can lead to serious errors in calculations. Therefore, the homogenous, thermal, gas phase decomposition of dry ozone was tried and found to be a satisfactory source if the residence time of ozone in the furnace is carefully regulated by furnace length and carrier gas flow, followed by rapid cooling of the products of decomposition.			

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Atomic Oxygen Thermal Ozone Decomposition Upper Atmosphere						

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